CEMENT
AND
CEMENT MANUFACTURE

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Specification for Cement with Low Heat Generation During Hydration.

IN the construction of the Pine Canyon Dam, in the United States, the specification for the cement contains provisions for the use of cement conforming to special requirements as to heat generation while setting. The dam will be 325 feet high and 275 feet thick at the base, and will contain 450,000 cubic yards of concrete.

The requirements for the cement include: (1) the cumulative heat of hydration must not exceed 65 cal. per gramme of cement at the age of seven days, and 80 cal. at 28 days; (2) tricalcium aluminate must not exceed 6 per cent. by weight; and (3) the fineness is to be 85 and 98 per cent. on the 200-mesh sieve. Standard-mortar compression specimens are to show 2,000 lb. strength at 28 days, but the 28-day strength is to be at least 35 per cent. higher than the seven-day strength.

Consideration of heating phenomena and the resulting stresses in this mass of concrete led to the preparation of special specifications for the cement. From a considerable group of special types of cement originally suggested, final specifications were prepared for three types of cement. The first was for standard A.S.T.M. specification cement, while the two others were for special cements.

The second alternative was for a special cement, called type B, conforming to the requirements of the standard specifications for Portland cement (serial designation C9-30) of the American Society for Testing Materials, with the following exceptions and additions: the residue on a standard 200-mesh sieve shall not exceed 15 per cent. by weight, nor be less than 2 per cent. by weight. The cumulative heat of hydration of the cement shall not exceed 77 cal. per gramme of cement up to the age of seven days after mixing with water, and shall not exceed 90 cal. per gramme of cement up to the age of 28 days after mixing with water.

The third alternative was a special cement, referred to as type C, which was required to conform to the requirements of the standard specifications for Portland cement (serial designation C9-30) of the American Society for Testing Materials, with the following exceptions and additions: (1) The percentage of tricalcium aluminate shall not exceed 6 per cent. by weight, when computed from the cement analysis by the method of Bogue as published in the Analytical Edition, *Industrial and Engineering Chemistry*, Vol. 1, No. 4, p. 192, Oct. 15, 1929. (2) The residue on a standard 200-mesh sieve shall not exceed 15 per cent. by weight, nor be less than 2 per cent. by weight. (3) No requirement shall be placed upon the tensile strength of standard mortar briquettes. (4) The average compressive strength of not less than three cylindrical test pieces, 2 in. in diameter and 4 in. in length, composed of mortar containing one part cement and three parts standard Ottawa sand, by weight, when tested in accordance with the tentative standards for compressive strength of Portland cement mortars (serial designation C9-16T) of the American Society for Testing Materials, shall be equal to or higher than the following:

Age at Test, Days.	Storage of Test Pieces.	Compressive Strength, lb. per sq. in.
7	1 day in moist air at 70 deg. F. 6 days in water at 70 deg. F.	800
28	1 day in moist air at 70 deg. F. 27 days in water at 70 deg. F.	2,000

The average compressive strength of standard mortar shall be at the age of 28 days at least 35 per cent. greater than the strength at the age of seven days. (5) The cumulative heat of hydration of the cement shall not exceed 65 cal. per gramme of cement up to the age of seven days after mixing the cement with water, and shall not exceed 80 cal. per gramme of cement up to the age of 28 days after mixing with water.

The cement is to be supplied at a rate not exceeding 2,000 barrels a day. The specifications require that no cement shall be despatched from the mill until it has passed the 28-day tests specified.

The following delivered prices were quoted by cement manufacturers: \$1.56 per barrel in bulk, or \$2.16 (\$1.76 net) in sacks, for type A, and \$1.65 in bulk, or \$2.25 (\$1.85 net) in sacks, for types B and C. Delivery was to be at the site in bins provided by the contractor.

In view of the advantages of the use of a cement with a heat of hydration that will be about two-thirds that of standard cement at seven days, and the resulting effect on the concrete mass during the process of setting and subsequent cooling, type C cement was selected and will be used for the main mass of the structure. The price difference of nine cents per barrel was believed to be considerably less than the advantage to be obtained by using the special cement. According to the specifications, the special low-heat cement need not be used in thin sections or accessories to the main structure.

Determination of the heat of hydration by the heat-of-solution method is made as follows: samples of 300 grammes of the dry cement, representing composite samples from each 1,000-barrel lot, are taken and kept sealed until tested. The cement sample and 120 grammes of distilled water are mixed at a temperature of 70 deg. F., with mechanical stirring for five minutes, and then placed in equal amounts in four or more containers. Each container is sealed, stored at 70 deg. F. for one day, and then maintained at 100 deg. F. until tested. At the age of seven and 28 days the heat of solution of two specimens of the partly hydrated cement is determined by the heat-of-solution method in a given acid mixture. Also, two determinations of the heat of solution of the dry cement are made in an identical acid mixture not later than the date of the 28-day test on the hydrated samples. The heat of hydration at the age of seven days is determined by subtracting the average heat of solution of the two specimens of cement hydrated for seven days from the average heat of solution of the two specimens of dry cement. Similar determination is made for the heat of hydration at the 28-day period.

The heat of solution, either dry or hydrated, is determined by dissolving a known weight of the cement in a dilute nitric and hydrofluoric acid mixture and observing the resultant temperature rise. The companies supplying the special cement are the Riverside Cement Co., the South-Western Portland Cement Co., the California Portland Cement Co., and the Monolith Portland Cement Co.

The same problem of avoiding changes of volume and consequent cracking in large masses of concrete has been considered in connection with the construction of the Hoover Dam in the United States, and cement with low-heat generation is also being used on this work. Mr. J. L. Savage, chief designing engineer for the Hoover Dam, states that two cements were considered, and a plant was erected to blend them where desirable. This blending plant will permit type A (low-heat) cement to be combined with type B (moderate-heat) cement in the proportions desired to meet different conditions, and will also permit the blending of cements of the same type coming from different mills to eliminate differences in composition, colour and temperature, and to produce a mixture of cements having essentially uniform workability, setting, hardening, strength and heat characteristics. The specifications embrace only true Portland cement.

Two types of cement are specified—one of low-heat evolution (type A) and one of moderate-heat evolution (type B).

Low-Heat Cement.

In the case of type A cement it is provided that the limits on chemical composition shall not exceed the following: loss on ignition, 3 per cent.; insoluble residue, 0.5 per cent.; sulphuric anhydride (SO_3), 2 per cent.; magnesia (MgO), 4 per cent.; uncombined lime (CaO), 1.5 per cent.; ratio of percentage of iron oxide to percentage of aluminium oxide, 1.5. The specified limits on theoretical compound composition, when computed according to the method outlined by Bogue, are: dicalcium silicate ($2CaO.SiO_2$),

not over 60 per cent.; tricalcium aluminate ($3CaO.Al_2O_3$), not over 5 per cent. It is contemplated that the final specifications may also impose upper limits on tricalcium silicate ($3CaO.Al_2O_3$) and on tetracalcium aluminoferrite ($4CaO.Al_2O_3.Fe_2O_3$).

The specification provision expressing fineness of the cement in terms of specific surface, with a range from 1,300 to 1,700 sq. cm. per gramme, is founded on a recognition of the inadequacy of the standard sieve test as a fineness indicator. The specific surface limits stated are based on determinations using micrometer apparatus and correspond in a general way to commercial Portland cement finenesses of 87 and 97 per cent. respectively passing the 200-mesh sieve The finenesses are not likely to be changed in the final specifications, although the specified values for specific surface may be altered to express them in terms of measurements by turbidimeter apparatus. The relatively high fineness range for the low-heat cement will improve the strength characteristics at early ages and accelerate the heat evolution.

The specifications include the customary standard test for soundness, with the proviso that in the final specifications there may be substituted, for the neat cement pat, a neat cement and/or mortar bar, approximately 1 in. square by 6 in. long, to permit length measurements before and after exposure to the steam. It is stipulated that initial set shall be developed in not less than 1 hour 45 minutes when the Gillmore needle is used, and that final set shall be attained within 10 hours. The use of the Vicat needle is eliminated.

The tensile strength test on 1 : 3 standard mortar briquettes is replaced by a compressive strength test on 3-in. by 6-in. concrete cylinders made with the same aggregate as is used in the work graded to ¾ in. maximum size. The concrete is to consist of one part of cement and 5.2 parts of aggregate by weight, with sufficient water to produce a fixed consistency corresponding to a 3-in. slump in a cone 10½ in. high. It is required that the 3-in. by 6-in. test cylinders, under standard conditions of curing, develop minimum strengths of 1,000 and 2,000 lb. per sq. in. at seven and 28 days respectively, and that the strength at 28 days be at least 50 per cent. higher than that at seven days.

Cumulative heat of hydration, as determined by the heat-of-solution method, is limited to 60 calories per gramme of cement at the age of seven days and 70 calories at 28 days. A further provision is that the temperature of the cement at the time of delivery shall not exceed 135 deg. F. during the months of May to September inclusive.

Moderate-Heat Cement.

The detail requirements for type B cement are the same as for type A cement with the following exceptions : no limit is placed on dicalcium silicate ($2CaO.SiO_2$) ; the limit on tricalcium aluminate ($3CaO.Al_2O_3$) is 8 per cent. ; the tricalcium silicate ($3CaO.SiO_2$) is limited to 60 per cent. The range of fineness is from 1,200 to 1,600 sq. cm. per gramme, corresponding roughly to finenesses of from 85 to 95 per cent. passing the 200-mesh sieve. The minimum compressive

strengths are 2,000 and 3,000 lb. per sq. in. at seven and 28 days respectively, and the strength at 28 days must be higher than at seven days. The cumulative heat of hydration must be greater than 70 calories per gramme at seven days and less than 100 calories at 28 days. There is no limitation on the temperature of type B cement, since it is not intended that this cement will be used where the minimising of concrete temperature is a primary objective.

The apparatus for measuring specific surface is a recent development of the U.S. Bureau of Standards. It is essentially a turbidimeter in which a light of constant intensity is passed through a suspension of the cement sample into a sensitive photo-electric cell, the current generated in the cell being measured with a microammeter. After certain characteristics of a given brand of cement have been established, the specific surface of a cement sample can be determined in a few minutes from a single reading of the microammeter. The gradation or size distribution of the cement particles can also be determined, if desired, from a succession of readings taken during the settling of the suspended material.

Heat of hydration is to be obtained by determining the heats of solution, in an acid solvent, of dry cement and partially hydrated cement and subtracting the latter from the former value. The apparatus to be employed is a heat-of-solution calorimeter developed by the Riverside Cement Co. It consists essentially of a calorimeter vessel in which the cement sample is dissolved and a calorimeter, both maintained at constant temperature. The calorimeter vessel is equipped with an electric heater coil, an electrical resistance thermometer, and a mechanical stirrer for agitating the liquid in the calorimeter as the cement is dissolved. The heat of solution is computed from recorded observations of the time-temperature rise of the liquid (during the period of dissolution) and a knowledge of the calorimeter constants. A period of 30 to 60 minutes is required for a heat-of-solution test.

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Influence of Phosphorus in the Raw Materials for Cement Manufacture.

By HENRI MARTIN (BRUSSELS).

It is strange to observe that the great majority of the textbooks on Portland cement manufacture do not refer to the part played by phosphorus in the raw materials, although other impurities such as alkalis, magnesia, and sulphuric anhydride are always commented upon. The matter is important, however, and certain manufacturers have paid dearly for their ignorance in this connection.

In the case under notice, phosphorus combined with lime occurred in certain layers of chalk in the form of tricalcic phosphate—a very stable compound and quite undecomposed by heat except under certain conditions, such as in the presence of silica and carbon at the temperature of the electric furnace, or, in other words, in the presence of silica under reducing conditions at very high temperature. These conditions are absent in the rotary kiln where the temperature is lower and the atmosphere is oxidising. Consequently it follows that lime combined with oxide of phosphorus in the state of $3CaO.P_2O_5$ cannot combine with the silica, alumina, and iron oxide of the clay in the raw material mixture.

Phosphatic chalk is of the type known to geologists as Turonian, a hard chalk having a yellowish-grey appearance, irregularly striated with brown streaks of iron oxide and speckled with very small dull grains of glauconite. It is thus easily recognisable, and in distinction to this, white chalk of the Senonian series contains no phosphorus.

It may appear extraordinary that in the chemical analysis of samples from a chalk deposit the presence of phosphorus can be undetected, but the reason is that, if the precaution of making a special test for this element is neglected, the phosphorus is contained in the precipitate of alumina and iron oxide; and not only is it ignored in the analysis, but it falsifies the determination of the alumina and iron oxide. It is therefore desirable that the examination of a deposit intended for cement manufacture should include a special test for phosphorus.

A method for the determination of phosphorus which has given excellent results is as follows: Boil 1 grm. of the material for 10 minutes with 40 ccm. of concentrated HCl diluted with an equal volume of water. Filter and add to the filtrate 100 ccm. of a solution composed of 100 grm. $MgCO_3$, 400 grm. citric acid, and 600 ccm. ammonia, made up to 1,500 ccm. with water. (When preparing this solution the ammonia should not be added until the reaction between the citric acid and $MgCO_3$ is complete and all CO_2 dispersed.) After addition of the 100 ccm. of this solution, add ammonia drop by drop until alkaline, then make strongly ammoniacal by adding 50 ccm. of concentrated ammonia. Stir vigorously until after a time a precipitate of magnesium ammonium phosphate is formed, then allow to stand for 12 hours before filtering, washing with 20 per cent. ammonia solution, and calcining to $Mg_2P_2O_7$.

Taking as an example a phosphatic chalk containing 5.44 per cent. P_2O_5 and 47.86 per cent. CaO, there would be present 11.87 per cent. tricalcic phosphate not decomposable in the rotary kiln, and thus leaving only 41.37 per cent. of lime available to combine with argillaceous elements. This corresponds to only 73.88 per cent. $CaCO_3$, which is much less than the requirement for a cement slurry. Such an example shows that, even though the impurity may remain inert in the finished cement, its presence cannot be ignored in the raw materials.

Obituary.

The death has occurred of Mr. E. W. G. Brooks, managing director of the Rugby Portland Cement Co., Ltd., at the age of 61 years.

The Hardening and Corrosion of Cement.—III.

By DR. KARL E. DORSCH
(Of the Technical High School, Karlsruhe, Baden).

The Viscosity of Cement during Setting.

The viscosity arising from the internal friction of the particles affords a criterion of the degree of gel formation in a system. The internal friction becomes greater as gelatinisation continues, and attains a maximum on prolonged hardening of the gel. Since in the setting of cement an originally fluid system gradually becomes viscous and finally quite hard, viscosity measurements will give an accurate representation of the setting and hardening process. The importance of viscometric methods for studying the kinetics of colloidal reactions was pointed out by Wo. Ostwald a considerable time ago. His work on the viscometry of gypsum suspensions and flour paste may be here mentioned.[1] He drew attention to the desirability of measuring the viscosity of setting cement, but did not undertake such measurements.

Viscometric measurements of a technical nature are now made in every laboratory in which cement is tested.[2] The setting-time test with the Vicat needle consists of measurements of viscosity by means of a needle of specified cross-section and load. This method is, however, only of technical value. It cannot be used in scientific work, since, as Platzmann[3] has recently shown, it gives insufficiently accurate values that are individually not reproducible.

The researches of Ostwald, like those of Maeda,[4] relate to very attenuated systems or suspensions, and thus realise the conditions which hold during the microscopical study of the hydration of small quantities of cement in the presence of large excess of water. But, as shown, the cement mortars of practice represent completely different conditions. The viscosity of technical cement pastes was first measured by H. Gessner,[5] using pastes containing 30 to 32 per cent. of water instead of the 26 to 27 per cent. required for normal consistency. He sucked the cement paste and a comparison liquid (*e.g.*, glycerine) through capillary tubes of the same diameter under considerably reduced pressure and measured the quantities flowing through during equal periods. The results were expressed in curves which show a slow increase of viscosity up to the beginning of setting, followed in the case of most cements by a much more rapid increase after a further half hour.

Gessner's results, according to which the setting of cement is accompanied by a continuous and ever more rapid increase in viscosity, appear at first sight to be not entirely in agreement with Nacken's[6] work on the hydration of cement.

(1) Wo. Ostwald and Wolski, *Kolloid Zeits.*, Vol. 27, p. 78, 1920; Ostwald and Lüers, *Ibid.*, Vol. 25, p. 117, 1918.
(2) L. Tetmayer, Communications of the Building Materials Testing Institute, Zurich, No. 5, p. 48, 1893.
(3) R. C. Platzmann, *Zement*, Nos. 37 and 43, 1929.
(4) T. Maeda, Sci. Papers Inst. Phys. and Chem. Research, Tokyo, Vol. 4, p. 102, 1926.
(5) H. Gessner, *Kolloid Zeits.*, Vol. 36, 1928: Vol. 37, 1929.
(6) R. Nacken, *Zement*, Nos. 47, 48, 1929; No. 16, 1927.

According to the latter the hydration of setting cement does not proceed continuously, but there are definite halts during the early period. These halts should also be evident in the changing viscosity of the cement-water system in view of the sensitivity of viscometric measurements.

The present author, in collaboration with A. Deubel, investigated the conditions under which the viscometric measurement of such highly viscous liquids as cement pastes is possible with the usual methods, and the extent to which these methods must be modified for such measurements. As a result, an accurate method for use with setting cement has been developed and applied in studying the effect of additions of electrolytes on setting.

Method of Measurement.

The most important condition in viscometry is absolute constancy of temperature. Since our apparatus was too large to work in a water thermostat, a

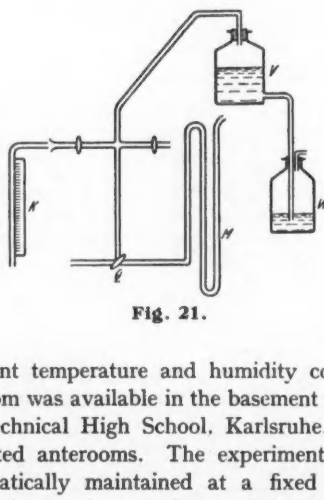

Fig. 21.

room in which constant temperature and humidity could be maintained was necessary. Such a room was available in the basement of the Building Research Department of the Technical High School, Karlsruhe. Access to this room is through two insulated anterooms. The experimental room is heated electrically and is automatically maintained at a fixed temperature by means of a thermoregulator. It was thus possible to maintain the complete apparatus at a constant temperature of 15 ± 0.2 deg. C. during the entire series of experiments. The temperature of the cement and mixing water was also thermally regulated.[7] The humidity in the room was maintained constant at 100 per cent. by means of a large dish of water several square metres in area in the anteroom. The experimental room also contained a shaking machine the speed of which could be kept constant by adjusting a resistance in conjunction with an ammeter.

For the measurements a modified Hess viscometer[8], shown diagrammatically in Fig. 21, was used. A slightly reduced pressure was first obtained in the vacuum

(7) Probst and Dorsch, *Zement*, No. 7, 1930.
(8) W. Stauf, *Kolloid Zeits.*, Vol. 37, p. 400, 1925.

flask V by means of a water pump, the pressure being read on the carbon tetrachloride manometer M. Water is thus drawn into V from the lower flask W, and when it has reached a certain level the water pump is cut out by means of the three-way cock Q. The reduced pressure is then proportional to the difference in height of the water surfaces in V and W. The combination of flasks V and W enables the pressure to be kept constant over a period, which is essential for a rapid series of measurements. The capillary tube is attached to the apparatus by the ground-in joint above K. Behind the capillary tube is a millimetre scale to enable the height attained by the viscous liquid to be read. Readings are taken at intervals of half a minute.

Before the apparatus is used for measurements during the setting of cements it must be calibrated, and its suitability for use with liquids of such high viscosity must be established. For example, if the results of the measurements were not reproducible the apparatus could not be considered satisfactory. The calibration is first carried out with liquids of various viscosities. Solutions of glue, gelatine, and casein in formamide are very convenient, since these three substances are soluble in formamide in all proportions to form liquids of paste-like consistency over a wide range of viscosity. After it was ascertained that the apparatus was completely satisfactory and capable of rapid working the investigation of the viscometry of mixtures of water and cement was begun. The first few experiments showed that it was essential to ensure that the mixtures were extremely homogeneous. This was attained by using cement of defined sieve fractions and by vigorously shaking the cement-water mix for ten minutes in the shaking machine.

It was next found that two factors made satisfactory measurements impossible. The first was segregation of the system due to sedimentation, and the second the development of a definite structure in the cement-water mix. The segregation effect increases with the water content of the mix. This effect is emphasised if the cement paste is allowed to stand for a time after mixing with water. If the capillary tube is immersed to a definite depth in the mix at the beginning of a series of tests, this sedimentation will cause totally different viscosity values to be obtained according to whether the measurement is made immediately or after some time has elapsed. If the end of the capillary tube is in the upper layers of the mix the earlier viscosity values are greater than those obtained later, since the heavier particles sink more rapidly and the lighter particles are left in the upper layers. If the capillary tube dips deeper into the mix the viscosity increases with time, since the heavy particles collect in the lower layers. In short, we are dealing with an entirely non-homogeneous, continuously changing system.

This applies not only to the motionless cement-water mix, but also to the fluid moving upwards under the influence of suction. In this case the extent of the sedimentation depends on the proportions of cement and water, the velocity of the liquid in the capillary (*i.e.*, the value of the reduced pressure), and the viscosity of the mix. Obviously the conditions are very complex, and controlled by many factors which are, for the most part, entirely unadjustable, so that reliable measurements are not possible.

How, then, can this sedimentation effect be eliminated? It cannot be entirely removed, but it can be minimised by immersing the capillary always to the same depth, accurate to a fraction of a millimetre. By this means we are always working with accurately determined liquids. The sedimentation in the capillary, which depends on the rate of flow, is, however, entirely a matter of chance, and cannot be quantitatively regulated. This sedimentation in the capillary under reduced pressure will always be evident to visual observation as a segregation of materials; after some time the uppermost part of the capillary will be seen to contain nothing but water.

The second disturbing factor is the development of a definite structural formation of the mix. According to the views of Michaelis, Kühl, and Nacken, a gel is at once formed when water and cement are brought into contact. This gel formation leads to structural development which renders accurate viscometric measurements impossible. This structure is destroyed by shaking and by inserting the capillary only at the instant of measurement. If the capillary is allowed to remain fixed in the cement paste for only a short time structural formation again sets in, leading to a stoppage in the tube.

The occurrence of this phenomenon led Gessner to state that setting cement showed the phenomenon of thixotropy.[9] True thixotropy cannot, however, occur in cement, since, on repeatedly shaking cement with water, new electrolyte is continuously formed, which has the greatest influence on setting and hardening; the cement continuously changes, and is finally completely decomposed as a result of the chemical disintegration of its constituent compounds.

But structure formation, like sedimentation, occurs in the capillary as well as in the mass of cement, and ultimately leads to stoppage. Its effect is seen when the continuous flow of the viscous liquid through the capillary suddenly slows up without external cause, and finally ceases, while water only—and not the water-cement mix—is sucked up the tube. The rapidity with which the water passes through depends on the density of the structural formation. If an attempt be made to avoid the formation of structural material by shaking the mixture during the experiment, as recommended by Ostwald, the viscosity value obtained is that for turbulent conditions, and not that for cement setting under conditions of rest.

The effect of sedimentation and structure formation was determined for all the cement-water mixes viscometrically investigated. Water additions of 30 to 33 per cent., as recommended by Gessner, proved unsuitable owing to pronounced segregation. In such concentrations the speed of separation was relatively greater than the speed with which the material moved in the capillary. Ostwald has previously shown that measurements on gypsum suspensions are only valid when the rapidity of separation is smaller than the speed through the capillary.

For our work we tried all known forms of viscometer, but in every case these difficulties arose and measurements were not possible. Even the falling-ball viscometer could not be used because the viscosities to be measured were too great,

(9) Freundlich and Rosenthal, *Kolloid Zeits.*, Vol. 37, p. 129, 1925. Freundlich and Rawitzer, *Kolloidchem. Beih.*, Vol. 25, 1926.

and further, the ball could not be seen owing to the opacity of the material. For these reasons results could not be obtained, in spite of the many variations of form of viscometer, pressure, and concentration investigated, even with the most homogeneous mixtures obtainable, and it therefore became necessary to develop a new method for the viscometry of such highly viscous materials. Accordingly an exact method was evolved which enabled the kinetics of setting cement to be quantitatively evaluated.

The viscometer used consists of a fine capillary tube of 0.1 mm. diameter, the immersed end of which carries a small interchangeable filter. This filter allows only the liquid sol to pass into the tube. The process depends on the following principles. During the setting of cement the denseness of its structure increases owing to the formation of structural gel. As the denseness increases it becomes more and more difficult to withdraw the liquid sol from the cement paste by

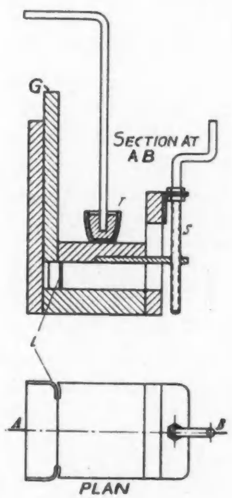

Fig. 22.

externally applied reduced pressure; this liquid sol is partially held within the gelatinous envelopes by capillarity, and partially exists in the interstices of the structure. The gel structure thus acts as a filter having pores of different sizes. As the denseness increases the speed with which the sol can be sucked out diminishes, and this rate of movement under suction can be measured. The increase in denseness can be expressed in terms of viscosity, which varies inversely as the speed in the capillary under reduced pressure. Ostwald and Rath[10] point out a similar relationship between two quantities which are not directly allied in their work on clay slips. Their method of stagalmometric drop formation cannot,

([10]) Ostwald and Rath, *Kolloid Zeits.*, Vol. 36, p. 243, 1925.

however, be used with technical cement pastes. The questions as to the extent to which this relationship between rate of movement in the capillary and viscosity holds good, and to what extent the hydration of cement can be expressed in terms of this movement require further research.

This immersion filter method which we have developed has numerous advantages. There is, for instance, no stopping up of the capillary tube, the cleaning of which—formerly very difficult and taking considerable time—is now easy. Measurements on technical cement pastes can now be made with great accuracy, whereas all other viscometric methods are impossible owing to the high viscosity of the almost solid cement.

Experimental Procedure.

The details involved in making a measurement are as follows. A considerable quantity of cement (maximum 600 g.) was mixed with almost sufficient water to give a paste of normal consistency (25 to 27 per cent. water). In practice we used 1 to 2 per cent. too little water, since it is important that there should not be a layer of water on the mix after shaking. The mix was placed in a well-stoppered metal container which was vigorously shaken for five to ten minutes. After shaking, the homogeneous paste was filled into small porcelain crucibles (T, Fig. 22) holding about 50 g. The crucibles were smoothed over so that they were just full. The amount of paste mixed suffices for twelve tests. The crucibles containing the cement paste were placed in position without shaking or disturbing them. In choosing the crucibles it must be remembered that in cement paste there are variations in viscosity not only in the vertical but also in the horizontal direction, and that these variations increase with the cross-sectional area of the containing vessel. This phenomenon can be detected when using the Vicat needle. It is accordingly necessary to use containers of the smallest possible height and diameter, and small porcelain crucibles were chosen for this reason.

Viscometric measurements were made on the cement paste in the crucibles at intervals of five to ten minutes. This was done by placing the crucible T,

TABLE 2.

	Normal Portland Cement. (Fig. 23).	High-strength Portland Cement. (Fig. 24).	Normal Portland Cement. (Fig. 25).	Aluminous Cement. (Fig. 26).
Loss on ignition ..	0.70	0.95	0.70	0.56
Insoluble residue ..	0.93	1.33	1.56	0.60
SiO_2	19.94	17.72	21.35	6.10
Al_2O_3	6.49	5.76	5.37	48.56
Fe_2O_3	3.53	3.96	3.76	10.44
CaO	63.44	65.83	63.57	34.04
MgO	2.33	1.16	1.08	0.18
SO_3	1.77	2.60	2.32	0.27

after a given time, underneath the capillary tube on a platform B, kept accurately in position by a vertical metal guide (G and L, Fig. 22), and capable of being raised at uniform speed in fifteen seconds by a screw S. The capillary tube is thereby immersed in the cement paste to a depth of 2.5 cm., accurate to 0.1 mm. The purpose of the immersing mechanism is to exclude the effect of variations in the manner of immersion. After standing for ten seconds the cock communicating with the vacuum flask V (Fig. 21) is opened and the liquid sol is drawn up into the capillary under constant reduced pressure. The height of the liquid in the capillary is measured at intervals of thirty seconds, and the results are plotted with the times during which the cement has been setting as abscissæ and the liquid heights as ordinates. Large liquid heights thus correspond to low viscosities.

TABLE 3.

Time of Setting in Minutes.	Normal Portland Cement. (Fig. 23.)	High-strength Portland Cement. (Fig. 24.)	Normal Portland Cement. (Fig. 25.)	Aluminous Cement. (Fig. 26.)
	Heights of liquid in capillary in centimetres.			
5	20.5	17.6	22.2	19.8
8	—	—	—	14.0
11	—	—	—	11.2
13	—	—	20.0	—
15	—	—	—	8.2
17	—	16.6	—	—
18	19.8	—	—	—
20	—	—	19.4	6.8
25	—	—	—	6.0
30	18.2	—	17.4	5.9
33	—	13.9	—	—
40	16.4	—	13.8	6.1
45	14.4	9.4	—	5.8
50	—	8.0	9.0	4.6
55	—	5.3	6.8	4.2
60	10.6	3.8	—	—
65	—	—	6.6	2.8
70	6.4	—	6.9	1.8
75	6.0	4.0	—	—
80	—	—	6.8	1.1
85	6.0	4.1	6.0	—
90	—	—	5.0	—
100	5.9	3.9	2.8	—
110	5.8	3.8	1.3	—
120	5.2	3.1	—	—
130	4.4	2.2	—	—
140	3.8	1.4	—	—
145	—	0.9	—	—
150	2.8	0.8	—	—
155	2.4	—	—	—

One high-strength and two normal Portland cements and an aluminous cement were investigated, the percentage chemical analyses being given in Table 2.

Results.

Table 3 gives a selection from the results obtained. The curves of Figs. 23 to 26 show a number of striking similarities between the cements investigated. All the curves show a characteristic break after about one hour, which marks the beginning of a period of constancy of the system lasting about thirty minutes. The action of water on the cement is most vigorous in the first hour or so, as is seen from the smaller slope of that part of the curve following the horizontal

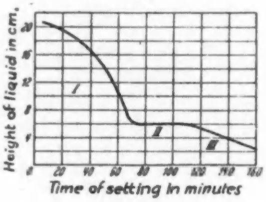

Fig. 23.

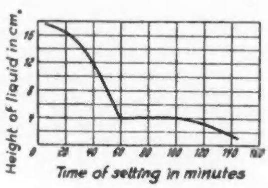

Fig. 24.

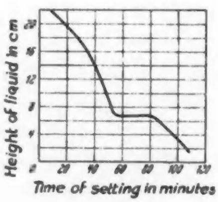

Fig. 25.

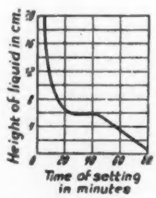

Fig. 26.

portion. With quick-setting cements such as aluminous cement there is a definite shortening of the horizontal portion of the curve which marks constancy of the system; this begins earlier, and the fall of the preceding part of the curve is more rapid. The curves may be interpreted by supposing that the reaction at once vigorously begins with copious and continuous gel formation, and that the clinker nuclei are finally protected by this gel from the further action of water. In other words, the initial violent reaction between water and cement, which surrounds the clinker grains with gelatinous envelopes, is marked by a steeply falling curve. The horizontal curve represents the clinker grains protected by these envelopes, which are finally broken down, so that water again has access to the clinker, which is again decomposed but more slowly than at first, and the curve falls less steeply. The curves so obtained from viscometric measurements are thus found satisfactorily to agree with the theory of Michaelis, Nacken, and Kühl.

This work related to technical cements, *i.e.*, to cements which had not been sieved. We next investigated the effect of different grain sizes on the viscometric setting curves of various cements. For this purpose the cements were separated into three sieve fractions: (1) not passing the 76 (per linear inch) sieve, (2) between the 76 and 180 sieves, and (3) passing the 180 sieve.

It was found that the coarse fraction neither set nor hardened. The medium and fine fractions gave similar curves to those in Figs. 23 to 26. The only distinction was that the fine material set somewhat more quickly than the medium, which was shown by the steeper initial curve and the earlier beginning of the horizontal portion in the case of the fine fraction.

The effect of temperature on the setting of the cements was also studied. It was expected that raising the temperature would accelerate, and reducing the temperature retard, setting as pictured by the new method. The viscometric measurements were made at 10, 20 and 30 deg. C., and the curves for these temperatures were found to run slightly apart and almost perfectly parallel. The 10-deg. curve was uppermost, with the 20-deg. and then the 30-deg. curves below. This result may be accepted as a confirmation of the correctness and accuracy of the method.

The method was finally used to investigate the effect of electrolytes on the viscosity of setting cement. Calcium chloride was chosen for the tests because of the technical importance of its accelerating action. The cements were mixed with water containing 0.5, 1, 2, 5 and 10 per cent. calcium chloride. Sideways-displaced parallel curves were obtained as in the study of the effect of temperature. Even the addition of 0.5 per cent. of calcium chloride to the mixing water showed a definite accelerating effect by this method, in contradiction to other recent results.[11] The difference between the results of other investigators and our own is, however, easily explained by the fact that calcium chloride varies in its effect with different cements.[12]

The author is at present working on a new method of measuring the viscosity of setting cement, which depends on changes in acoustical and mechanical vibrations and their measurement electrically. This has the advantage that there is no disturbance of the viscous medium so that continuous measurements can be made on the same sample. An account of this work will be given later in a suitable periodical.

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(11) W. N. Thomas, Building Research Special Report No. 14, 1929.
(12) K. E. Dorsch, *Bauingenieur*, Nos. 11 and 12, 1930.

Cement Production in Italy.

The production of Portland cement in Italy was 288,776 tons in October 1932, 294,136 tons in October 1931, and 324,358 tons in October 1930. For the period January to October 1932 the production was 2,685,551 tons, against 2,666,227 tons and 2,998,330 tons for the same periods in 1931 and 1930 respectively.

Action of $CaSO_4$ on Ground Clinker.

EXPERIMENTS with precipitated gypsum, calcium sulphate hemihydrate and soluble anhydrite (prepared by heating the gypsum to 105 deg. C.) are described by P. Schachtschabel in a recent number of *Zement*. The experiments were made on a well-burnt rotary-kiln clinker ground to 21.4 per cent. residue on the 250 (per inch) sieve, 10 per cent. on the 180 sieve, and 0.1 per cent. on the 76 sieve. The ground clinker was sealed in metal boxes immediately after grinding. Without addition and with mixing for one minute the initial setting time was one minute and the final setting time two minutes ; the temperature rise was 10 deg. C. The cement and water were mixed for one minute and three minutes to study the effect of time of mixing. The results are given in Table I.

TABLE I.

VARIATION OF SETTING TIME DUE TO GYPSUM, HEMIHYDRATE AND SOLUBLE ANHYDRITE.

Water, 26 to 28 per cent. Times of Mixing, 1 and 3 minutes.

Per cent. Addition.	Gypsum.				Hemihydrate.				Sol. Anhydrite.			
	Mix 1 min.		3 mins.		1 min.		3 mins.		1 min.		3 mins.	
	Init. h. m.	Final. h. m.	Init. h. m.	Final. h. m.	Init. h. m.	Final. h. m.	Init. h. m.	Final. h. m.	Init. h. m.	Final. h. m.	Init. h. m.	Final. h. m.
0.75	—	—	—	—	—	—	—	—	0 3	0 4	0 2	0 3
1.00	0 3	0 4	—	—	0 3	0 5	—	—	4 15	6 45	5 25	8 20
1.25	0 2	0 6	0 9	0 12	4 40	6 40	4 50	8 0	—	—	—	—
1.50	0 8	2 50	1 45	3 30	—	—	—	—	—	—	—	—
2.0	2 4	5 50	3 30	5 30	4 50	7 35	5 0	7 50	4 50	7 30	5 0	7 40
2.5	6 20	9 7	5 15	8 0	0 45	6 30	4 30	7 20	—	—	—	—
3.0	—	—	—	—	0 29	6 0	3 45	6 45	0 34	5 50	1 42	6 35
4.0	6 45	10 0	5 25	8 25	0 25	5 25	0 27	5 45	0 33	5 0	0 29	5 7
6.0	—	—	—	—	0 17	0 33	0 16	0 30	—	—	—	—
8.0	6 0	10 30	5 55	8 40	—	—	—	—	—	—	—	—

Although the gypsum was very finely divided, one-half per cent. more was required to give normal setting time with mixing for one minute than for three minutes ; this shows the great effect of velocity of solution on setting time. Normal set could not be obtained (even with six minutes' mixing) with less than 1.5 per cent. of gypsum. With the same ground clinker stored in paper bags for three weeks (initial set, without additions, 14 minutes, final 48 minutes), one-half per cent. of gypsum gave normal setting time.

Using hemihydrate instead of gypsum, time of mixing had no effect owing to the great velocity of solution of hemihydrate. The use of 1.25 per cent. (corresponding in SO_3 content to 1.5 per cent. gypsum) gave normal setting time. With 2.5 per cent. and more the setting time was again reduced ; with 3 per cent. this can be prevented by longer mixing, but with 4 per cent. this is no longer possible. This phenomenon is due to the formation of a supersaturated solution of hemihydrate instead of its conversion in solution to the dihydrate ; this property is also common to soluble anhydrite. It can be prevented by adding a small amount of gypsum to the hemihydrate, which catalytically increases

the velocity of the change from hemihydrate to gypsum so that supersaturation does not occur.

"False set" is often found with hemihydrate and soluble anhydrite (*e.g.*, with 3 per cent. hemihydrate and mixing for one minute (see Table I). This is also due to the formation of supersaturated hemihydrate solution. Finally the change from hemihydrate to gypsum suddenly and vigorously proceeds so that solid gypsum is precipitated, mechanically carrying a large quantity of water with it. This removal of water causes the false set. After some time this gypsum is dissolved, the mechanically held water is set free, and setting proceeds normally.

That this false set is due to the mechanical removal of water is shown by mixing the ground clinker plus 4 per cent. hemihydrate with increasing quantities of water. The results with 24 per cent. water were initial set 25 minutes, final 5 hours 25 minutes; with 30 per cent. water, initial 3 hours 10 minutes, final 7 hours 25 minutes. The increase in water content rendered the cement slow setting.

The results with soluble anhydrite were similar to those for hemihydrate. Dehydration at 105 and 165 deg. C. gave the same results. 1 per cent. was necessary for normal set. The ground clinker, without addition, must not be too quick-setting. If it sets in one minute a slow-setting cement cannot be obtained by adding soluble anhydrite. This shows that the anhydrite must be converted to hemihydrate before it can dissolve and act as a retarder; the change requires two minutes in the presence of water. False set is also observed with the anhydrite, and can also be avoided by adding a small proportion of gypsum.

The view that rapid setting is due to greater temperature rise is not valid. Thus the temperature rise of ground clinker with 3.2 per cent. of soluble anhydrite or 3.4 per cent. of hemihydrate is 2 deg. C.; with 4 per cent. of gypsum it is 3.5 deg. C.

Table II shows the effect of various substances dissolved in the mixing water.

TABLE II.

EFFECT OF $CaCl_2$, $MgCl_2$, K_2SO_4 AND Na_2SO_4 ON GROUND CLINKER CONTAINING GYPSUM OR HEMIHYDRATE.

	2.5 p.c. Gypsum.				4 p.c. Gypsum.				1.25 p.c. Hemi-hydrate.		4 p.c. Hemi-hydrate.	
	Mix 1 min.		3 mins.		1 min.		3 mins.		1 min.		3 mins.	
	Init. h. m.	Final h. m.	Init. h. m.	Final h. m.	Init. h. m.	Final h. m.	Init. h. m.	Final h. m.	Init. h. m.	Final h. m.	Init. h. m.	Final h. m.
No addition ..	6 20	9 7	5 15	8 0	6 45	10 0	5 25	8 25	4 40	6 40	0 24	5 40
1 p.c. $CaCl_2$ ..	3 23	5 40	5 0	6 45	3 18	5 40	4 40	7 30	3 15	5 15	0 35	5 0
1 p.c. $MgCl_2$ ..	—	—	—	—	5 20	8 15	5 15	8 0	3 50	6 25	0 32	4 40
1 p.c. K_2SO_4 ..	5 25	8 25	4 40	8 55	2 10	7 10	1 55	6 0	4 30	7 0	0 10	5 0
1 p.c. Na_2SO_4 ..	6 10	9 0	5 20	8 10	6 50	8 50	5 35	8 10	4 30	6 50	0 39	6 0

One per cent. $CaCl_2$ accelerates, except in the case of 4 per cent. hemihydrate when there is a slight retarding effect. The acceleration with mixing for one

minute was greater than with three minutes' mixing. The amount of gypsum used did not affect this acceleration. The accelerating action of 1 per cent. $MgCl_2$ is less than that of 1 per cent. $CaCl_2$.

According to the literature of the subject K_2SO_4 has no effect on setting time ; Table II shows that this is incorrect, and that it has an accelerating action which is greater with the higher gypsum content and with the longer mixing time. Na_2SO_4 is without effect on gypsumised cement.

Both Na_2SO_4 and K_2SO_4 are without effect with 1.25 per cent. hemihydrate. With 4 per cent. hemihydrate K_2SO_4 accelerates and Na_2SO_4 retards set. The difference from gypsum is due to double salt formation. The results are the same with soluble anhydrite.

The effect of alkali carbonates in the mixing water was also studied. They have an accelerating action due to their conversion of the gypsum into $CaCO_3$. Na_2CO_3 has a greater effect than K_2CO_3. Increasing the gypsum and keeping the carbonate constant converts a quick-setting into a slow-setting cement ; or, with more gypsum, more carbonate can be added before the cement becomes quick setting. The effect of time of mixing and different amounts of water with gypsum and carbonate additions was investigated. Increasing the mixing time retards setting (*e.g.*, one minute mixing, initial set five minutes, final set 15 minutes ; five minutes mixing, initial set 54 minutes, final set three hours). Water content plays a subordinate part.

The action of alkali carbonates with 2 per cent. hemihydrate is the same as for 3 per cent. gypsum. With 4 per cent. hemihydrate the effect is different. K_2CO_3 progressively accelerates, while Na_2CO_3 first retards and then, with increasing additions, accelerates ; 0.8 per cent. Na_2CO_3 gives the longest setting time (initial 1 hour 8 minutes, final, 2 hours 8 minutes). Na_2CO_3 can thus prevent false set. The difference is again due to double salt formation.

With soluble anhydrite, alkali carbonates have a much greater accelerating action than with hemihydrate, and the retarding action of small Na_2CO_3 additions does not occur. This is due to the delay of two minutes in the change from anhydrite to hemihydrate.

Cement Production in the United States.

The U.S. Bureau of Mines states that the production of Portland cement in the United States amounted to 72,261,000 barrels for the period January to November 1932, compared with 118,596,000 barrels and 152,425,000 barrels for the same period of 1931 and 1930 respectively. The percentage of production to capacity was 29.1 for November 1932, 37.2 for November 1931, and 51.7 for November 1930. The percentage of production to capacity for 12 months ending November 1932 was 29 per cent., compared with 44.4 for twelve months ending November 1931, and 62.6 per cent. for twelve months ending November 1930. In each of the three years, 165 plants were in operation.

Compression and Transverse Tests of Portland Cement.

By A. C. DAVIS, M.I.Mech.E., M.Inst.C.E.I., F.C.S.

WORKS MANAGING DIRECTOR, ASSOCIATED PORTLAND CEMENT MANUFACTURERS, LTD.

THERE is frequent discussion of the desirability or otherwise of compression tests, as an alternative to the tensile tests hitherto adopted in the British Standard Specification for Portland Cement. In the matter of cement quality, the tensile test of standard sand mortar is still the one upon which reliance is placed in ascertaining whether the cement is satisfactory or not. There are, however, so many factors which determine the strength of concrete that if it is desired to

Fig. 1.

get an indication of what this will be tests must be made with the actual ingredients which will be used, and this can best be done by a compression or transverse test. For this purpose it is also important that the mixing, tamping, and storage of the specimens should be conducted under the practical conditions which will occur when the concrete is made and used, but, within the limits imposed by those conditions, procedure should be standardised as much as possible.

Compression Tests.

The compression test of 3 : 1 standard sand mortar is specified in the cement specifications of about half the countries of the world which have adopted official

standards for cement. The minimum resistance required varies between 1,700 and 3,200 lb. per sq. in. at seven days and between 2,000 and 5,000 lb. at twenty-eight days ; these wide limits are due in the main to varying ideas of consistency and methods of making and storing specimens.

As frequently carried out in this country by cement manufacturers as a check on the strength of their product, the test for compression or crushing is commonly performed on a cube of 3 : 1 standard sand mortar of 50 sq. cm. area (about 2¾ in. cube). Cylinders are sometimes used, but they appear to possess no great advantage, while they have a disadvantage in the actual crushing operation.

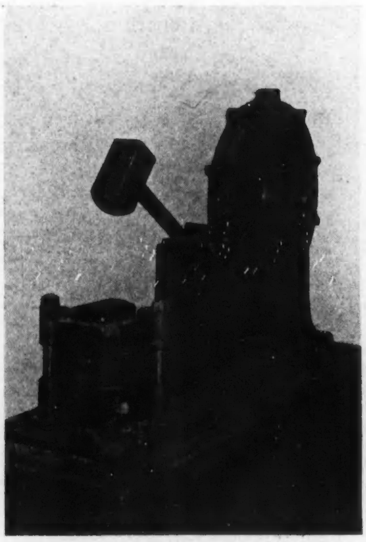


Fig. 2.

It has been said that the " order of merit " into which different cements may fall when tested for tensile strength in standard 3 : 1 mortar does not always agree with their order when tested in concrete. This argument is put forward as a reason for the abandonment of the tensile test in the British Standard Specification for Portland Cement and the substitution therefore of a compression or transverse test, either of the same grade of mortar or of a concrete mix. The explanation of this alleged unsatisfactory feature of the tensile test of standard mortar may be found to lie in the same direction as the objections raised against the now almost obsolete tensile test of neat cement, namely, that a 3 : 1 mix, while better than a neat paste, is still too rich in cement to bring out to the full those adhesive and cohesive qualities which are of the first importance in constructional work which demands the use of mixes containing less cement.

As with the making of tensile tests, it is necessary to use uniform and controlled procedure for the preparation of the compression test blocks in laboratory practice. Standard mortar blocks for compression are made by well mixing 250 grammes of cement with 750 grammes of dry standard sand. To this is added about 80 c.c. of clean water (or the same quantity as for the standard briquettes), and the mortar well stirred and rammed into an accurately machined metal mould. The British Standard spatula for making mortar briquettes is useless for this purpose, and it is usual to use a block of wood or metal which just fits into the mould, and to hammer this either by hand or machine (see Figs. 1 and 2).

After the preparation of the block by this process, the top and bottom faces are trowelled off and the specimen put aside to set for a period of 24 hours under

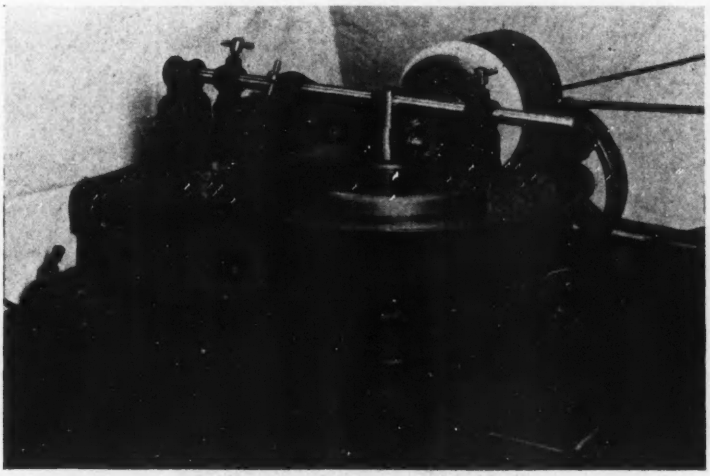


Fig. 3.

the standard humidity and temperature conditions for briquettes, afterwards being placed in water, as with briquettes. The immersion lasts until the test takes place, and each block must be tested immediately it is taken from the water—in short the procedure follows the British Standard Specification for tensile tests.

In the Standard Specifications of those countries which have officially adopted the compression test the procedure is usually somewhat as follows (this being a free translation from the German normal specification):

" 500 grammes of cement and 1,500 grammes of standard sand are first mixed dry by hand for one minute with a light spoon in a basin. Then 160 grammes (8 per cent.) of water are added to the dry mix. The damp mass is mixed for another minute, then evenly distributed in a mortar mixer (Fig. 3) and mixed

by twenty turns of the machine. Next, 860 grammes of the mortar mixed as above are placed in a mould provided with a receiving box, and beaten by 150 blows of the hammer apparatus (Fig. 2). After taking away the receiving box the mortar remaining is smoothed with a knife.

"The cubes are placed in receptacles in their moulds and are kept damp. They are taken out of the moulds after about twenty hours; and twenty-four hours after they are made the cubes are placed in water with a temperature of 17 deg. to 20 deg. C. The water must cover the cubes to be tested by at least 2 cm., and must be renewed every fourteen days.

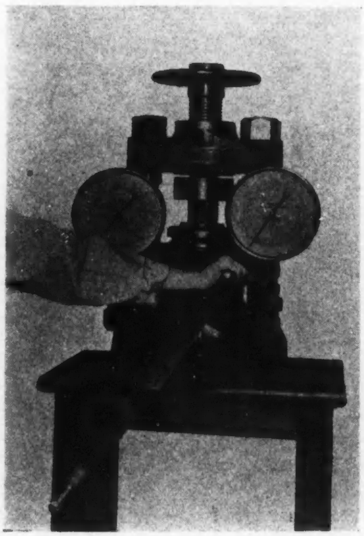


Fig. 4.

"Test cubes which are to be hardened in air must stand free and separately on three-edged wooden supports or some similar arrangement, and be stored in a closed room or cupboard free from draught. The temperature of the room should be from 17 deg. to 20 deg. C., and the relative air humidity should be 55 per cent. to 80 per cent.

"Test cubes which are hardened under water should not be taken out of the water until immediately before the test is to be made.

"The pressure indication of the machine to be used for the test of crushing strength should be exact to at least 1.5 per cent. The highest pressure obtained is reckoned as the crushing strength of the material tested. As the rate at which pressure is applied has an influence upon testing results, care should be taken

that the pressure is increased at an average rate of 20 kg. per square centimetre per second.

" The average of results from separate tests (as a rule five) is decisive for the crushing strength. Faulty tests are not to be included. The pressure is always to be tested on the side surfaces of the cube, not on the bottom and upper surfaces which have been worked upon."

Machines for carrying out compression tests are usually hydraulically operated, oil being used as the pressure medium instead of water (see Fig. 4). Other types of machines of the necessarily robust and accurate design generally cost more, and they may be slower to manipulate and take up more room.

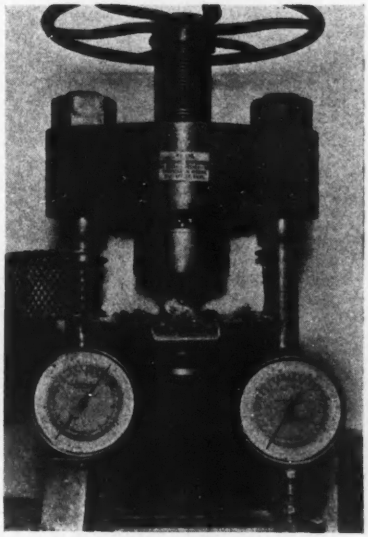


Fig. 5.

In these machines one or both of the compression plates rests on a spherical seating so as to ensure a uniform bearing on the specimen. This is very important. The upper compression plate is almost invariably carried by a screw spindle, which is used to set the distance between the plates to the size of the test block. During the test the upper plate is at rest, whilst the lower plate is raised by the piston pressing upwards under the action of oil coming from the plunger pump, which is driven by hand or electric motor. The pressure of oil in lb. per square inch in the main cylinder multiplied by the sectional area of the piston in inches is equal to the total strain on the specimen block provided that the pistons move without friction, and this is usually arranged in the construction of the apparatus. The oil also assists to

lubricate and tighten the piston. The pressure exerted acts at the same time upon a pressure gauge or mercury column, and the accuracy of the results read off depends on the proper working of the pistons in their cylinders. If the machine is designed without a stuffing box or other lining for tightening the pistons, the friction will be smaller rather than greater when the machine is loaded because of the slight widening of the cylinder.

As regards accuracy, a good hydraulic machine is equal to a good lever machine ; in respect to wear and tear, it is almost indestructible, in fact, the machine will improve by frequent use in consequence of the wearing away of any roughness on the cylinders and pistons. No special foundation is normally required for hydraulic crushing machines, save only that the floor should be strong enough to carry the weight.

Fig. 6.

Fig. 7.

As previously mentioned, a very important point to be noted in testing cubes or cylinders by compression is the manner in which the specimens are bedded on the compression plates. It is necessary that the distribution of the load over the top and bottom surfaces of the block should be quite uniform. Cylinders may be bedded by using plaster of Paris. The plaster is levelled on the two ends to a thickness of about a quarter of an inch, and when it is set the test may be made. Leather, linoleum, lead and other forms of packing are also used. Alternatively the ends may be ground flat and parallel. It is, however, usually considered preferable to make cubes in metal moulds with plane and parallel sides of such accuracy that the specimens may be crushed on two of the sides which have been in contact with the mould without any packing, and this simple and convenient way of securing an even bedding in the testing machine is a strong point in favour of cubes as compared with cylinders. A typical failure of a concrete cube tested to destruction under these conditions is shown in Fig. 5.

The compression strains which result from a mixture of cement and sand, or cement, sand and stone, may vary within very wide limits due to the proportions of the mix ; the amount of water used in mixing ; thoroughness with which the mixing is conducted ; method of consolidation in the moulds ; size, shape and grading of the aggregate ; physical structure of the aggregate ; construction of the mould ; size, shape and age of the specimen ; method of storage; method of testing ; and temperature and atmospheric conditions.

In dealing with compression tests made of concrete being used in constructional work, all these points must be kept in mind. The precise control which is obtainable in a laboratory is usually lacking in such cases, and, therefore, such tests are inherently less reliable and must be viewed more tolerantly.

Figs. 6 and 7 illustrate two of the most popular methods of consolidating concrete in the moulds for the purpose of obtaining specimens of some uniformity. In Fig. 6 a metal punner of a known weight, with a face of a specified area, is used, and the concrete filled into the mould in three or more equal layers, each of which receives the same number of blows with the punner. In Fig. 7 the punner is substituted by a bullet-nosed rod of specified diameter, and the procedure is very similar to that adopted with the punner. The method to be used depends to some extent upon the nature and consistency of the mix, and the following figures of tests will serve to illustrate the importance of adapting the method of consolidating the concrete to consistency of the material, or *vice-versa.*

Compression Tests of Laboratory-made Concrete, using Washed and Graded Thames Ballast and Sand (uncrushed).

	Results in lb. per sq. inch.											
	6 : 3 : 1 Mix. Moulds filled by				4 : 2 : 1 Mix. Moulds filled by				3 : 1½ : 1 Mix. Moulds filled by			
	Punning.		Rodding.		Punning.		Rodding.		Punning.		Rodding.	
	7 days.	28 days.	7 days.	28 days.	7 days.	28 days.	7 days.	28 days.	7 days.	28 days.	7 days.	28 days.
Dry mix	2,259	4,812	1,820	2,717	5,257	6,303	2,740	4,150	5,623	7,644	3,855	5,277
Medium mix ...	2,507	3,167	1,762	3,073	3,196	4,568	3,330	4,694	3,900	6,322	3,836	6,271
Wet mix	2,073	3,000	1,928	3,119	2,824	4,435	2,923	4,830	3,797	5,462	4,069	5,523

All specimens were 6in. cubes, and the results are the average of three specimens in each case.

It will be seen that the specimens which were punned into the moulds gave the strongest concrete, but they decreased in strength as the quantity of water was raised, whereas when the concrete was compacted by rodding the strength tended to rise with the more workable concrete. With wet concrete there was little to choose in strength between the two methods of compacting.

As regards the quality of aggregates and their effect on the strength of concrete, the recommendations of the Institution of Structural Engineers are as follows :—

Fine Aggregate.

" The fine aggregate should consist of hard siliceous grains, crushed stone, or other approved material. It should be cleaned, and free from injurious

amounts of clay and any animal, vegetable, bituminous, or other deleterious matter. Unless initially clean and free from dust, all fine aggregate should be thoroughly washed with clean water. All fine aggregate should be capable of passing through a mesh $\frac{3}{16}$ of an inch square measured in the clear, and not more than 10 per cent. should pass through a sieve having 40 meshes to the linear inch and wires of 0.0125in. diameter, and it should be well graded between these limits. The fine aggregate should be separated from the coarse aggregate before the materials are measured."

Coarse Aggregate.

"The coarse aggregate should be clean sea, river or pit gravel, or should consist of broken stone of hard nature, or other equally hard suitable approved

Fig. 8.

material, not flaky. The following materials should not be used with the fine or coarse aggregate in the composition of the concrete: (*a*) Coal residues (including clinkers, cinders, ashes, coke breeze, pan breeze), clay, or other similar material; (*b*) Blast-furnace slag, copper slag, forge breeze, dross or other similar materials; (*c*) sulphates, including plaster of Paris and other similar materials; (*d*) broken bricks, unless they are approved.

"The coarse material should be of such a size as will pass through a mesh $\frac{3}{4}$in. square measured in the clear, and be retained on a mesh $\frac{3}{16}$in. square measured in the clear, and should vary in size as much as possible within these limits. For heavy reinforced construction, such as rafts, the maximum size recommended may be increased to one-fifth of the minimum space between any two pieces of reinforcement, or between reinforcement and shuttering. The maximum size

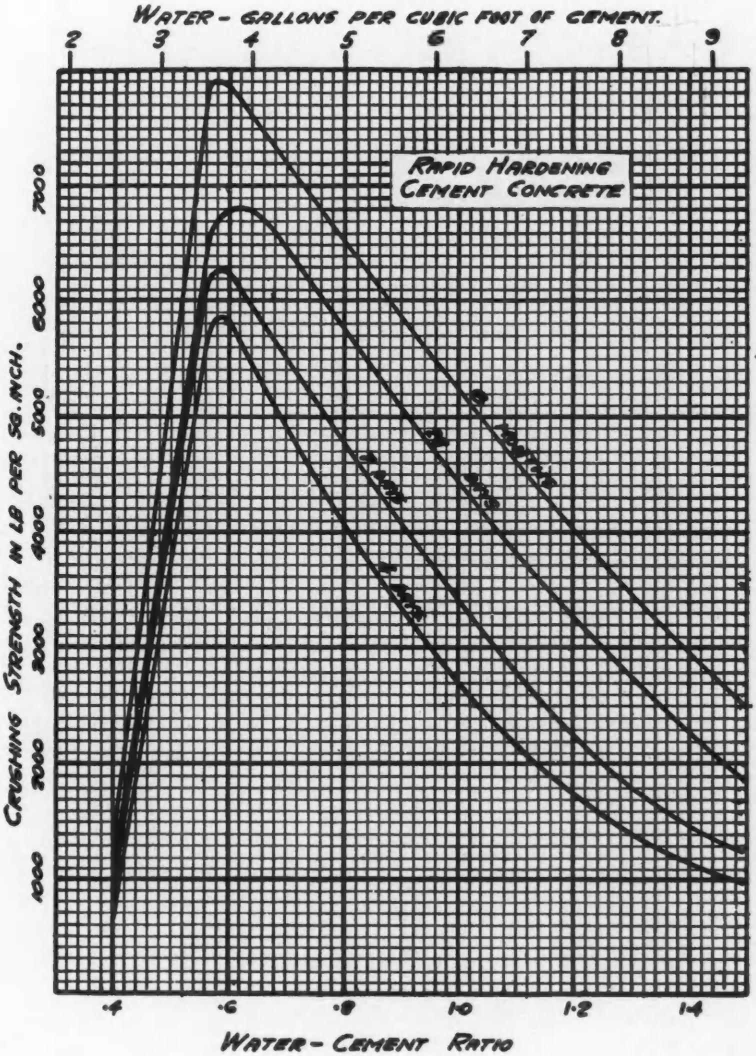

Water - Gallons per cubic foot of cement.
2
3
4
5
6
7
8
9
Rapid Hardening
Cement Concrete
1000
2000
3000
4000
5000
6000
7000
Crushing Strength in lb per sq. inch.
·4
·6
·8
1·0
1·2
1·4
Water - Cement Ratio

Fig. 9.

of aggregate for such portions of concrete as are free from reinforcement may exceed the limits suggested above. For sea or river work, pipes, tanks, and all structures exposed to or to contain water or other liquids, special care should be taken to avoid the use of aggregate of a porous nature. Unless it is quite clean, coarse aggregate should be thoroughly washed with clean water.

"When so required, and before the work is commenced, laboratory tests should be made of the aggregates to be used on the work to establish their suitability for concrete, and from time to time check tests should be made of deliveries to the site to ensure that uniformity is being maintained."

In ordinary commercial machine-mixed concrete made with four parts by measure of coarse aggregate, two parts of fine aggregate (as defined in these recommendations), and one part of cement gauged to a plastic consistency (but not too wet) a crushing strength of about 3,000 lb. per sq. in. or more can be expected in one month. These tests, however, usually vary so much for reasons discussed earlier that the precise figure will vary within wide limits.

A more useful guide will perhaps be the following results of some field tests of concrete used in connection with various public works, which afford some idea of the results obtained under ordinary conditions in civil engineering work with present-day cements.

In making compression tests from concrete taken from the mixer it is of value to record the "slump" by the method now very generally adopted and which may be shortly described as follows. The apparatus needed is a sheet-metal truncated-cone mould, 12in. high, 8in. diameter at the base, and 4in. diameter at the top, provided with handles at the sides (see Fig. 8), and a $\frac{5}{8}$in. bullet-nosed metal rod 1ft. long. The mould is filled with concrete in layers of 4in. at a time, each layer being prodded thirty times with the rod. After the third and last layer has been rodded and levelled, the mould is lifted off and the slump measured. Tests show that Thames ballast concrete having a slump of from $\frac{1}{2}$in. to 1 in. will contain only a little more water than is necessary for maximum strength. Care must be taken to ensure that the concrete being tested is not caused to slump by vibration of the board on which it stands when the mould is removed.

The test should be applied to a portion of the first batch of concrete that is mixed, and if the slump is not in accordance with the specification further tests should be made with varying quantities of water. Having arrived at the correct consistency, the test should then be applied several times a day to ensure that the required consistency is uniformly maintained. A competent inspector or foreman can easily control the quantity of water, and therefore the strength of the resulting concrete. Excess of mixing water may decrease the strength of concrete as much as if some of the cement content had been left out. The quantity of mixing water must be kept as small as practicable for the class of work being undertaken. The same consistency should be used in all batches for the same class of work.

Specimens taken from	Mix	Slump	Age	Crushing strength lb. per sq. in.		Method of curing, etc.
				6in. cubes	6in. by 12in. cylinders	
		inches	days			
Reinforced concrete road.	4 parts Kent ragstone, ¾in. to ¼in., 1 part Thames sand, and 1 part ordinary Portland cement.	—	12	2,750 2,800 2,700		
Reinforced concrete bridge, No. 1.	1 : 1 : 2 (pit ballast, sand, ordinary Portland cement).	1¾	28	6,050 6,250 5,950 5,850 6,400 6,100	4,160 3,962 4,358 4,002 4,002 4,358	Cubes and cylinders taken from moulds after 2 days and then completely immersed in water for 7 days. Specimens then left in air until due for test.
Do. do.	Do. do.	2½	28	5,150 5,000 4,900 5,250 4,850 5,550	4,754 4,952 4,556 4,556 4,754 4,635	
Do. do.	Do. do.	4 (Water-cement ratio 41%.)	28		3,724 3,684 3,724 4,041 4,160 4,120	
Do. do.	Do. do.	½	28		3,962 3,645 4,358 3,566 4,991 4,912	
Reinforced concrete bridge, No. 2.	1 : 2 : 4 (Thames ballast, sand, ordinary Portland cement).		28	2,850 3,000 3,400		Cubes left on site for 24 hours under damp sacking, then placed in damp sand.
Do. do.	1 : 3 : 6 (do.).		28	950		
Do. do.	1 : 2 : 4 (Thames ballast, sand, rapid-hardening cement).		28	4,450 4,300		
Do. do.	1 : 1½ : 3 (do.).		28	3,450 3,600		
Reinforced concrete bridge, No. 3.	1 : 2 : 4 (pit ballast, sand, ordinary Portland cement).	2 to 3	14	1,500 2,050 2,750		

Specimens taken from	Mix	Slump	Age	Crushing strength lb. per sq. in.		Method of curing, etc.
				6in. cubes	6in. by 12in. cylinders	
		inches	days			
Reinforced concrete bridge, No. 3.	1 : 2 : 4 (pit ballast, sand, ordinary Portland cement).	2 to 3	28	3,100		
				2,900		
				3,000		
				2,650		
				3,650		
				3,330		
				2,950		
				2,850		
				2,850		
				3,000		
				3,400		
				4,050		
				3,000		
Do. do.	Do. do.	2 to 4	28	3,000		
				2,825		
				2,700		
				2,250		
				2,400		
				2,350		
				3,550		
				3,000		
				3,800		
				3,500		
				2,900		
				3,250		
				4,050		
				3,650		
				4,025		
				4,000		
				3,650		
				3,900		
				3,200		
Do. do.	1 : 3 : 6 (pit ballast, sand, ordinary Portland cement).	2 to 4	28	1,200		
				1,000		
				1,800		
				1,800		
				1,250		
				1,500		
				1,800		
				2,000		
				1,700		
				1,500		
				1,550		
				1,500		
				1,950		
Do. do.	Do. do.	2 to 4	90	2,950		
				2,850		
Do. do.	1 : 2 : 4 (pit ballast, sand, ordinary Portland cement).	2	7	1,300		
				1,300		

Specimens taken from	Mix	Slump	Age	Crushing strength lb. per sq. in. 6in. cubes	6in. by 12in. cylinders	Method of curing, etc.
		inches	days			
Reinforced concrete bridge, No. 3.	1 : 2 : 4 (pit ballast, sand, ordinary Portland cement).	2	28	3,250 2,950 2,600 2,750 3,150 3,000 3,100		
Do. do.	1 : 2 : 4 (pit ballast, sand, rapid hardening cement).	Wet mix.	7	2,000 2,400		
Do. do.	Do. do.	Do.	28	3,600 3,050 3,400 3,300 3,000 2,650 3,100 3,100 2,750 2,350 3,050 2,200 2,250		
Reinforced concrete pier.	1 : 1.6 : 3.2 (4 cu. ft. crushed washed ballast, ¾in. to ¼in., 2 cu. ft. washed sand, 112 lb. rapid-hardening cement).	2	7	3,250 3,350 4,625 4,150 4,350 3,950 3,800 4,550 4,650 3,725 3,675 3,775		
Do. do.	Do. do.	2	28	6,200 6,600 5,900 5,350 5,450 5,100 4,900 4,675 4,600 4,950 4,950 5,100		

NOTE.—Specimens were taken throughout the year under prevailing weather conditions, and temperatures at the time of test were not recorded.

The following slumps have been recommended for different types of concrete using Thames ballast aggregate :

Class of Concrete.	Maximum Slump (in.)
Mass concrete	2
Reinforced concrete :—	
Thin vertical sections	6
Heavy sections	2
Thin confined horizontal sections	8
Roads and pavements :—	
Hand finished	4
Machine finished	1
Mortar for floor finish	2

So long as tests are being made of a given mix of concrete, using the same aggregate, it is possible to make very useful comparisons of the consistency of the concrete by this method. It fails, however, to afford comparative results when varying mixtures, and more especially aggregates of varying character, are used. A convenient way of specifying the amount of water to be used is to determine the ratio of water to cement (or cement to water) which gives the desired consistency with the aggregates and proportions specified. Fig. 9 shows the relation between water-cement ratio and strength for concrete made with rapid-hardening cement, and a similar relationship obtains for ordinary Portland cement.

In practical work there should be no difficulty in securing that the concrete will give the desired crushing strength by the proper control of the work, and especially of the proportions and consistency.

The ratio between the tensile and crushing strains of concrete is not invariably constant. The compressive strength increases more rapidly with age than does the tensile strain.

Transverse Tests.

Little is now heard respecting transverse tests of Portland cement mortar and concrete, although the test is used for concrete products such as roofing tiles and paving slabs. Investigations have been made from time to time to determine whether this test is useful as a laboratory check upon the quality of cements, but it has yet to be shown conclusively that the test offers advantages over the tensile strength test.

It has at times been suggested that a transverse test should be incorporated in cement specifications, but this test would appear to have a greater sphere of usefulness in the testing of mortars and concretes whose composition makes them unsuitable for making up into the small briquettes necessary for the tensile test. The transverse testing of cement quality (as distinct from that of cement products) has therefore not received much official recognition, although for many years it has been used in an intermittent and experimental way. Its chief claim to consideration is perhaps the ease with which such a test can be conducted without expensive machines, and the latitude it allows for variation of the dimensions of the

specimens to be tested. It sometimes happens, moreover, that the transverse test is the only available method of testing, particularly with regard to concrete articles of the type mentioned, where a tensile or crushing test is clearly impracticable.

A very simple arrangement of two knife edges upon which the article is to rest, together with a means of applying the load to the centre of the unsupported span of the article, can be made to give reliable results. In a case where the weights applied are multiplied by a lever arrangement, care must be taken that the load is applied perpendicularly and at right angles to the axis of the bar.

The relation between the transverse test and tensile test of a given sample of cement varies as much with regard to conditions as does the relation between the tensile and crushing tests. In testing concrete the cross-section of the bar must depend to a large extent upon the size of the aggregate used, to avoid the possibility of any large piece of stone taking up a considerable proportionate area of the point under stress.

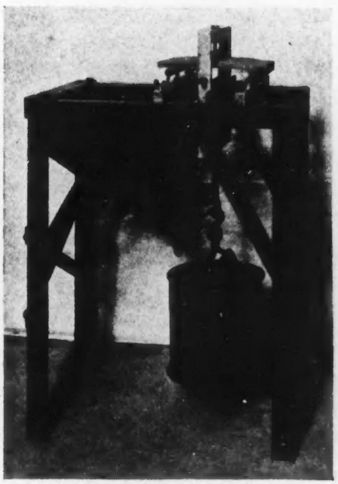


Fig. 10.

Fig. 11.

Transverse tests of small bars of standard mortar are somewhat unreliable unless carried out by a careful and experienced tester under properly controlled conditions, and, as already stated, the test finds its most useful field in determining the strength of small factory-made articles, such as roofing tiles, whose dimensions and shape prevent either a tensile or crushing test. Figs. 10 and 11 show the method of making such a test on roofing tiles.

It has been stated that the difference in the times of setting, and of the early hardening of rapid-hardening cements, is a factor that must be correctly gauged so as to avoid unnecessary delay due to waiting longer than is necessary for the

hardening of concretes after they have been placed in position. It is, for instance, not only important for the contractor to know how long he may work his cement without damage, but also how long it takes to obtain sufficient strength to permit the removal of forms and so on. The difference in the times of hardening of various cements and the important bearing this has upon the strains to be applied to "green" concrete can be accurately gauged by the use of strength tests only by very exact control of all conditions.

In both the tensile and compression tests results will not be obtainable before the cement has set hard, because whilst in the "green" state immediately after the final set the test blocks will not bear any appreciable strain; but results are ascertainable within 24 hours of the final setting of the cement for most present-day cements of a rapid-hardening character.

German Cement Sales.

The sales of the Deutscher Zement Bund for the month of November 1932 were 178,000 tons, compared with 195,000 tons in November 1931 and 317,000 tons in November 1930. For the eleven months January to November the sales were 2,694,000 tons in 1932, compared with 3,609,000 and 5,297,000 tons for the same period in 1931 and 1930 respectively.

The Hochofenwerk Lubeck A.G. Herrenwyk in Lubeck reports a loss of RM.1,946,206 (£97,310) for 1931–32, compared with a net profit of RM.1,300,000 (£65,000) for 1930–31.

Cement Production in Canada.

The production of Portland cement in Canada in July 1932 was 457,246 barrels, compared with 1,111,253 barrels in July 1931. For the period January to July 1932 the production was 2,764,893 barrels, against 5,649,697 barrels for the same period in 1931.

Cement Production in Belgium.

For November 1932 the production of Portland cement in Belgium was 49 per cent. of total capacity, compared with 48 per cent. for November 1931. For the period January to November 1932 production was 41.98 per cent. of capacity, compared with 51 per cent. for the same period of 1931.

Cement Manufacture in Brazil.

A recent Decree prescribes conditions under which certain privileges will be granted to enterprises manufacturing cement in Brazil from raw materials of national origin. Exemption from Customs duties, "expectiente" and other taxes during a period of ten years is granted to enterprises producing a minimum of 25,000 tons of cement a year for machinery, apparatus, tools, instruments and materials. These exemptions apply only to the installation, extension and alteration of works and services, including the replacement of parts, but do not cover any material which enters into the composition of cement, containers, fuel or lubricants, and other material used in operation, or any merchandise similar to existing national products.

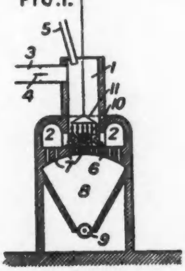
Recent Patents relating to Cement.

Cements.

373,248. Pontoppidan, C., 33, Vestergade, Copenhagen. Feb. 13, 1931.

In the manufacture of cement in a rotary kiln, the granular or pulverulent charge material is preheated and wholly or partly calcined before delivery to the kiln, in a

FIG. I.



chamber (1) through which other waste gases from the kiln and the charge material are passed alternately. The chamber (1) is fitted with heat-absorbing bodies such as chains (10) which, as shown, are carried by a vertically movable beam (11) so that they may be lowered for heating up during the passage of the hot gases from the supply ducts (2) to the outlet flue (3), and raised through the charge material to impart heat thereto. A damper (4) is provided in the flue (3) for cutting off the flow of gases before the raw material is fed to the chamber (1) from the chute (5); in some cases the passage of hot gases may be continued during transfer of heat from the chains to the raw material. The bottom (6) of the chamber (1) and ducts (2) is formed with perforations (7) through which the preheated material passes to a chamber (8) fitted with a conveyer (9) delivering to the rotary kiln. The chains (10) may be replaced by grates or by ribs or inclined surfaces down which the material slides by gravity. Continuous operation may be secured by the employment of a pair or a number of pairs of alternately operated preheating chambers.

Cements: Filtering.

380,239. Dorr Co., Inc., 247, Park Avenue, New York, U.S.A. Aug. 28, 1931.

A small quantity of lime, or other flocculation increasing agent, is added to cement

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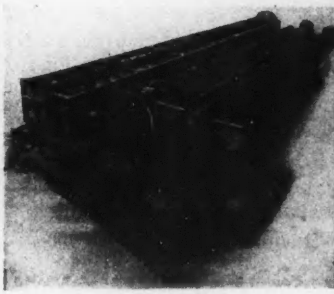

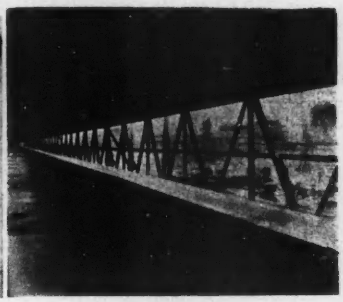

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or marl slurry to facilitate filtration. The amount of lime or the like is less than 1 per cent., and is preferably about 0.1 per cent.

Porous Cement.

373,792. Fox, J., Rock Cliffe, Westminster Road, Eccles, and Harrap, E. R., 53, Brook Road, Chorltonville, both in Manchester. June 5, 1931.

Aluminium powder and soda ash are used as foam-producing chemicals in the production of porous cement for insulating material.

Cement Mortars and Concretes.

373,442. Case, G. O., Ellis, E. M., and Montigue, L. H., 115, Gower Street, London. June 11, 1931.

A composition for the manufacture of blocks, bricks, slabs, etc., comprises limestone, bathstone, Portland stone, Kentish rag or other rock containing a large proportion of calcium carbonate, crushed to such a degree of fineness that the whole passes a 30-mesh sieve and at least 50 per cent. passes a 50-mesh sieve, mixed with from 5 to 13 per cent. by weight of the total mixture of Portland cement, aluminous cement, etc. Aggregate such as gravel may be added.

Recent Patent Applications.

No. 381,223. H. E. White. Manufacture of Portland cement.

No. 384,060. H. A. Gill (F. L. Smidth and Co., Aktieselskab). Processes of and plant for burning cement, lime, and like materials.

No. 384,736. M. Vogel-Jorgensen. Method of and plant for wet grinding of raw cement material.

Cement Production in Portugal.

The production of Portland cement in Portugal was 9,630 tons in October 1932 compared with 7,316 tons in October 1931. For the period January to October 1932 the output was 100,408 tons, compared with 79,503 tons for January to October 1931.

NOTICE.

PAPER SACKS.

In an action in the Chancery Division brought against B. Kershaw & Co. (1920) Limited to restrain infringement of Bates' Patents Nos. 252038x, 250917 and 251016 protecting "open-mouthed" and "valved" sewn paper sacks and machinery for making same, the Defendants on the 9th December, 1932, submitted to an injunction in respect of all three patents.

The above patents are owned by Paper Sacks Limited of Northfleet, Kent, and it is their intention to enforce their rights against all persons attempting to infringe.

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Notes from Abroad.

Cement Quotas in Turkey.

It is reported that the quotas for Portland cement and white cement under the import restrictions now in force in Turkey for the period January to March 1933, are 450 tons and 135 tons respectively.

Belgian Cement Companies' Reports.

S. A. des Cimenteries et Briqueteries de Tilleur reports a net loss of Frs. 75,480 (£431) for the year ended June 30, 1932, compared with a net profit of Frs. 360,675 (£2,061) for the previous year.

S.A. des Ciments Meuse-Brabant.—The net loss for the year ending September 30, 1932, was Frs. 207,049 (£1,183), compared with a net profit of Frs. 1,308,479 (£7,477) for 1931, and Frs. 1,074,654 (£6,141) for 1930. The directors stated that cement consumption in Belgium for 1932 was 10 per cent. less than in 1931, and that whereas the selling price fell at the beginning of the year by about 35 per cent., the cost of production had only been progressively reduced by 20 per cent. The company's works at Orp-le-Grand and Riviere-Lustin had only been in operation for six and ten months of the year respectively.

German Cement Company's Report.

Hessische Portland Cementfabrik Steinau A.G. has closed the financial year 1931 with a loss of RM. 1,944 (£97) on a share capital of RM. 200,000 (£10,000).

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